भारतीय मानक
Indian Standard

IS 11913 : 2023

टर्ट-ब्यूटाइलहाइड्रोक्विनोन — विशिष्टि

(पहला पुनरीक्षण)

Tert-Butylhydroquinone (TBHQ) — Specification

(First Revision)

ICS 67.220.20

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Food Additives Sectional Committee, FAD 08

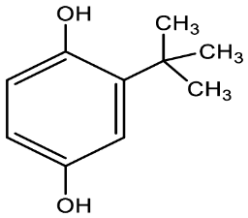
FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

Food additives are added to improve the appearance, flavour, texture or storage properties and in some cases to enhance the nutritive value of the processed foods. As certain impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. A series of standards has, therefore, been prepared by this Bureau to cover purity and identification of these substances. It is hoped that these standards would help in checking purity which requires to be checked at the stage of manufacture, for it is extremely difficult (and in many cases impossible) to detect the impurity once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by scientists, health authorities and international bodies.

tert-butylhydroquinone (TBHQ) is a white, crystalline solid having a characteristic odour. It is used as an antioxidant in edible oils and fats and whole milk powder in the country.

tert-butylhydroquinone is also known as mono-*tert*-butylhydroquinone, *tert*-butylquinol, and 2-tertbutyl-1,4-dihydroxybenzene. Its empirical formula is $C_{10}H_{14}O_2$. Its molecular weight is 166.22 and structural formula is:



The standard was first published in 1986 and in its preparation considerable assistance was derived from the *Food Chemical Codex, Published National Academy of Sciences and National Research Council, Washington DC, USA*.

In this revision, the following changes have been made:

a) The identification test for TBHQ has been aligned with corresponding JECFA Monograph;
b) The requirement for heavy metal (as Pb) is replaced by lead (as Pb); and
c) It also incorporates one amendment issued to this standard.

The composition of the Committee responsible for the formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

Tert–BUTYLHYDROQUINONE (TBHQ) —SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for *tert*-butylhydroquinone (TBHQ).

2 REFERENCES

The standards given below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

IS No.	*Title*
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 1699 : 1995	Methods of sampling and test for food colours (*second revision*)

3 REQUIREMENTS

3.1 Description

tert-butylhydroquinone shall be a white crystalline solid having a characteristic odour. It shall be soluble in alcohol and ether but practically insoluble in water.

3.2 Identification

Dissolve about 5 mg of the sample in 10 ml of methanol, and add 10.5 ml of dimethylamine solution (1 in 4). A red to pink colour is produced.

3.3 Melting Range

The melting range of the product shall be between 126.5 °C and 128.5 °C.

3.4 The material shall also conform to the requirements given in Table 1.

4 PACKING AND STORAGE

4.1 The material shall be filled in well-closed containers, so as to preclude air contamination of the contents with metal or other impurities.

4.2 Storage

The material shall be stored in a cool and dry place.

5 MARKING

5.1 Each container shall be marked legibly to give the following information:

a) Name of the material including the words ‘Food Grade’;
b) Source of manufacture;
c) Net quantity when packed;
d) Batch or code number;
e) Date of manufacture;
f) Instruction for storage;
g) Best before date (month and year to be given by the manufacturer); and
h) Any other requirements as specified under the *Legal Metrology* (*Packaged Commodities*) *Rules,* 2011 and *Food Safety* and *Food Safety and Standards* (*Packaging*) *Regulations*, 2018 *and Food Safety and Standards* (*Labelling and Display*) *Regulations,* 2020.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provision of *Bureau of Indian Standards Act*, 2016 and Rules and Regulation framed thereunder and the product(s) may be marked with the Standard Mark.

6 SAMPLING

The representative samples of the material shall be drawn and conformity of the material to the requirements of this specification shall be determined according to the procedure prescribed in IS 1699.

7 TESTS

7.1 Tests shall be carried out by the methods specified in co1 (4) of Table 1.

7.2 Quality of Reagents

Unless specified otherwise pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for *Tert*-Butylhydroquinone (TBHQ)

(*Clause* 3.4**)**

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Purity as $C_{10}H_{14}O_2$, percent by mass, *Min*	99	**A-1**
ii)	*t*-butyl-*p*-benzoquinone, percent by mass, *Max*	0.2	**A-2**
iii)	2,5-di-*t*-butylhydroquinone, percent by mass, *Max*	0.2	**A-3**
iv)	Hydroquinone, percent by mass, *Max*	0.1	**A-3**
v)	Arsenic (as As), mg/kg, *Max*	3	IS 1699
vi)	Lead (as Pb), mg/kg, *Max*	2	IS 1699
vii)	Toluene, mg/kg, *Max*	25	**A-4**
viii)	Ultraviolet absorbance (polynuclear hydrocarbons)	Passes test	**A-5**

ANNEX A

(*Clause* 3.4, *Table* 1)

METHOD OF TESTS FOR *tert*-BUTYLHYDROQUINONE (TBHQ)

A-1 PURITY

A-1.1 Reagents

A-1.1.1 *Methanol*

A-1.1.2 *Sulphuric Acid* — 1 N

A-1.1.3 *Diphenylamine Indicator* — three milligram of *p*-diphenylaminesulphonic acid sodium salt per ml of 0.1 N sulphuric acid.

A-1.1.4 *Ceric Sulphate* — 0.1 N

A-1.2 Procedure

Transfer about 170 mg of the sample, ground to a fine powder and accurately weighed, into a 250 ml wide-mouthed Erlenmeyer flask, and dissolve in 10 ml of methanol. Add 150 ml of water, 1 ml of 1 N sulphuric acid, and 4 drops of diphenylamine indicator. Titrate with 0.1 N ceric sulphate to the just complete colour change from yellow to red-violet. Record the volume, in ml of 0.1 N ceric sulphate required. If HQ and DTBHQ are present in the sample they will be included in the titration and the correction as given in the formula shall be applied.

A-1.3 Calculation

Purity as $C_{10}H_{14}O_2$, percent by mass (A) =

$$\frac{8.311 \times N\,(V - 0.1)}{M}$$

where

V = volume, in ml, of 0.1 N ceric sulphate required;
N = normality of the ceric sulphate solution; and
M = mass, in g, of the sample taken.

NOTE — 0.1 ml represents the volume of ceric sulphate solution consumed by the primary oxidation products of *tert*-butylhydroquinone ordinarily present in the sample.

A-1.3.1 If HQ and DTBHQ are present in the sample they will be included in the titration and the following correction shall be applied:

Purity, as $C_{10}H_{14}O_2$, corrected percent by mass = A — (percent HQ × 1.51) — (percent DTBHQ × 0.75)

A-2 *t*-BUTYL-*p*-BENZOQUINONE

A-2.1 Apparatus

A-2.1.1 *Spectrophotometer* — A double-beam infrared spectrophotometer with matched 0.4 mm liquid sample cells with calcium fluoride windows.

A-2.2 Reagents

A-2.2.1 *Monotertiary-butyl-p-benzoquinone Reference Standard*

A-2.2.2 *Carbon Tetrachloride*

A-2.3 Procedure

A-2.3.1 *Preparation of Standard*

Transfer about 10 mg of accurately weighed monotertiary-butyl- p-benzoquinone Reference Standard, into a 10 ml volumetric flask. Dissolve in carbon tetrachloride, dilute to volume with the same solvent and mix.

A-2.3.2 *Preparation of Sample*

Transfer about 1 g of the sample, accurately weighed and previously reduced to a fine powder in a high speed blender, into a 10 ml volumetric flask. Dilute to volume with carbon tetrachloride, and shake for 5 minutes to extract the t-butyl-benzoquinone. Filter through a millipore filter or equivalent before use.

A-2.3.3 Fill the reference cell with carbon tetrachloride and the sample cell with the standard preparation (**A-2.3.1**). Place the cells in the respective reference and sample beams of the spectrophotometer and record the infra spectrum from 1 600 cm^{-1} to 1 775 cm^{-1}. On the spectrum draw a background line from 1 612 cm^{-1} to 1 750 cm^{-1}, and determine the net absorbance (A_s) of the standard preparation at 1 659 cm^{-1}. Similarly, obtain the spectrum of the sample preparation (**A-2.3.2**), and determine its net absorbance (A_u) at 1 659 cm^{-1}.

A-2.4 Calculation

t-butyl-*p*-benzoquinone, percent by mass =

$$\frac{A_u}{A_s} \times \frac{W_s}{W_u} \times 100$$

where

A_u = net absorbance of the sample preparation;
A_s = net absorbance of the standard preparation;
W_s = exact mass, in mg, of the reference standard taken; and
W_u = exact mass, in mg, of the sample taken.

A-3 2, 5, Di-t-BUTYLHYDROQUINONE AND HYDROQUINONE

A-3.1 Apparatus

A-3.1.1 *Gas Chromatograph* — of a suitable type equipped with a thermal conductivity detector, containing a 0.61 m × 6.35 mm (O.D.) stainless steel column packed with 20 percent Silicone SE-30, by mass and 80 percent Diatoport S (60/80-mesh), or equivalent materials.

A-3.1.1.1 *Operating conditions*

The operating parameters may vary depending upon the particular instrument used, but a suitable chromatogram may be obtained using the following conditions:

a) *Column temperature* — programmed from 100 °C to 270 °C at 15 °C per minute;
b) *Injection port temperature* — 300 °C;
c) *Carrier gas* — helium or nitrogen flowing at a rate of 100 ml per minute;
d) *Bridge current* — 140 ma; and
e) *Sensitivity* — 1 × for integrator, 2 × for recorder.

A-3.2 Reagents

A-3.2.1 *Hydroquinone, 2,5-di-t-butylhydroquinone and Methyl Benzoate Stock Solution*

Weigh accurately about 50 mg each of hydroquinone (HQ) 2,5-di-t-butylhydroquinone (DTBHQ), and methyl benzoate (internal standard). Transfer into separate 50 ml volumetric flasks, dilute to volume with pyridine and mix.

A-3.3 Procedure

A-3.3.1 *Calibration Standard*

Into separate 10 ml volumetric flasks, add 0.50 ml, 1.00 ml, 2.00 ml and 3.00 ml of the HQ stock solution. Then to each flask add 2.00 ml of methyl benzoate (internal standard) stock solution, dilute each to volume with pyridine and mix. In the same manner prepare four DTBHQ calibrating solutions. Prepare the trimethylsilyl derivative of each solution as follows - add 9 drops of calibration solution to a 2 ml serum vial, cap the vial, evacuate with a 50 ml gas syringe, add 250 µl of N, O-bis-trimethylsilyl-acetamide, and heat at about 80 °C for 10 minutes. Chromatograph 10 µl portions of each standard in duplicate, and plot the concentration ratio of HQ to internal standard (X-axis) against response ratio of HQ to internal standard (Y-axis). Plot the same relationship between DTBHQ and the internal standard.

A-3.3.2 *Sample Preparation and Procedure*

Transfer about 1 g of the sample, accurately weighed, into a 10 ml volumetric flask. Add 2.00 ml of the methyl benzoate internal standard stock solution, dilute to volume with pyridine and mix. Prepare the trimethylsilyl derivative as described in **A-3.3.1** and then chromatograph duplicate 10 µl portions to obtain the chromatogram. The approximate peak times in minutes are: Methyl benzoate-2.5; trimethylsilyl derivative of HQ-5.5; trimethylsilyl derivative of *tert*-butylhydroquinone-7.3; trimethylsilyl derivative of DTBHQ-8.4.

A-3.4 Calculation

A-3.4.1 Determine the peak area (response) of interest by automatic integration or manual triangulation. Calculate the response ratio of HQ and DTBHQ to internal standard. From the calibration curves determine the concentration ratio of HQ and DTBHQ to internal standard, and calculate percent HQ and percent DTBHQ as follows:

HQ or DTBHQ, percent by mass =

$$r \times I \times \frac{10}{S}$$

where

r = concentration ratio (X-axis on calibration curve);
I = percent (w/v) of internal standard in the sample preparation; and
S = mass, in g, of the sample taken.

A-4 TOLUENE

A-4.1 Apparatus

A-4.1.1 *Gas Chromatograph* — of a suitable type equipped with a flame ionization detector containing a 3.66 m × 3.18 mm (O.D) stainless steel column packed with 10 percent Silicone SE-30, by mass, and 90 percent Diatoport S (60/80-mesh), or equivalent material.

A-4.1.1.1 *Operating conditions*

The operating parameter may vary depending upon the particular instrument used, but a suitable chromatogram may be obtained using the following conditions:

a) *Column temperature* — programmed from 70 °C to 80 °C at 15 °C per minute and held;
b) *Injection port temperature* — 275 °C;
c) *Carrier gas* — helium or nitrogen flowing at a rate of 50 ml per minute;
d) *Cell temperature* — 300 °C;
e) *Hydrogen and air settings* — 138 kPa each; and
f) *Sensitivity* — 1×10^2.

A-4.2 Reagents

A-4.2.1 *Toluene*

A-4.2.2 *Octyl Alcohol*

A-4.3 Procedure

A-4.3.1 *Preparation of Standard Toluene Solution*

Prepare a solution of toluene in octyl alcohol containing approximately 50 µg per ml and calculate the exact concentration (C_r) In percent (*m*/*v*).

A-4.3.2 *Preparation of Sample Solution*

Transfer about 2 g of the sample accurately weighed, into a 10 ml volumetric flask. Dissolve in octyl alcohol. Dilute to volume with the same solvent and mix. Calculate the exact concentration of the solution (C_S) in percent (*m*/*v*).

A-4.3.3 Inject a 5 µl portion of the standard solution into the chromatograph, and measure the height of the toluene peak on the chromatogram. The toluene retention time is 3.3 minutes. Similarly obtain the chromatogram on a 5 µl portion of the sample solution, and measure the height of the toluene peak. Calculate the percentage of toluene in the sample.

A-4.4 Calculation

$$\text{Toluene, mg/kg} = \frac{H_S}{H_r} \times \frac{C_r}{C_S} \times 10^6$$

where

H_s = height of the toluene peak of the sample solution;
H_r = height of the toluene peak of the standard solution;
C_s = concentration in percent (*m*/*v*) of the sample solution; and
C_r = concentration in percent (*m*/*v*) of the standard solution.

A-5 ULTRAVIOLET ABSORBANCE

A-5.1 Reagents

A-5.1.1 *L-Ascorbic Acid*

A-5.1.2 *Ethanol*

A-5.1.3 *Isooctane*

A-5.1.4 *Anhydrous Sodium Sulphate*

A-5.1.5 *Hexadecane*

A-5.2 Procedure

A-5.2.1 Dissolve 1 g of L-ascorbic acid in 100 ml of ethanol and 100 ml of water contained in a 500 ml separator (S-1). Transfer about 50 g of the sample, accurately weighed into the separator. Shake to dissolve, then add 50 ml of isooctane and extract for 3 minutes. After the phases have separated, drain the lower aqueous phase into a second 500 ml separator (S-2), then after 1 minute of further separating, drain the lower layer into the separator (S-2). Add a second 50 ml portion of isooctane to the aqueous solution in S-2 and repeat the extraction procedure as previously described, drawing off the lower, aqueous layer into a third 500 ml separator (S-3). Add a third 50 ml portion of isooctane to the aqueous solution in S-3 and repeat the extraction procedure as previously described, drawing off and discarding the lower aqueous layer.

A-5.2.2 Extract each isooctane solution (that is, the solution in S-1, S-2, S-3), with two 100 ml portions of a 0.5 percent solution of ascorbic acid in ethanol-water (25 : 75). Shake each mixture for 1 minute, allow the phases to separate, and discard the lower, aqueous layers. Next, extract each isooctane solution with two 100 ml portions of a 5 percent solution of ethanol in water, and discard the lower, aqueous layers. Finally, wash each solution twice with 100 ml of water, and discard the washes.

A-5.2.3 Lightly pack a standard size chromatographic tube with 100 g of anhydrous sodium sulphate, and wash the packed column with 75 ml of isooctane, discarding the wash. Filter the isooctane solution from S-1 through the column, and collect filtrate in a 500 ml distillation flask. Wash S-1 with the isooctane solution contained in S-2, and then pour the solution onto the column, collecting the filtrate in the flask. Wash S-2 and S-1, successively, with the iso-octane solution in S-3, and filter the solution through the column as before. Wash S-3, S-2, and S-1 in that order and in tandem with two successive 25 ml portions of isooctane, and

pass the washings individually through the column and into the flask. Let the column drain completely.

A-5.2.4 Add 2 ml of hexadecane and 2 boiling stones to the 500 ml distillation flask containing the combined isooctane extracts, and attach the flask to a suitable vacuum distillation assembly. Evacuate the assembly to about one-third atmosphere, then immerse the flask in a steam bath, and distill the solvent. When isooctane stops dripping into the receiver, turn off the vacuum, wash down the walls of the flask with 5 ml of isooctane added through the top of the distillation head, then replace the

thermometer and again evacuate. The isooctane should distill over in about 1 minute. At the end of this distillation add another 5 ml portion of isooctane and repeat the stripping procedure.

A-5.2.5 Quantitatively wash the residue from the distillation flask into a 50 ml volumetric flask with isooctane, dilute to volume with isooctane, and mix. Determine the ultraviolet absorption spectrum of the solution in a 5 cm silica cell from 400 mμ to 250 mμ, with a suitable spectrophotometer, using isooctane as the blank. Determine the absorbance of a solvent control by following the above procedure in every detail, but with the sample omitted. From the sample spectrum determine the maximum absorbance per cm path length in each of the following wavelength intervals:

a) 280 mμ to 289 mμ;
b) 290 mμ to 299 mμ;
c) 300 mμ to 359 mμ; and
d) 360 mμ to 400 mμ.

Calculate the maximum net absorbance per cm in each interval by subtracting from the sample absorbance the corresponding absorbance per cm of the solvent control. The following net absorbance values are not exceeded at the indicated intervals: (a) 0.15; (b) 0.12; (c) 0.08; and (d) 0.02.

ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

Food Additives Sectional Committee, FAD 08

Organization	*Representative(s)*
CSIR - Indian Institute of Toxicology Research, Lucknow	DR YOGESHWAR SHUKLA **(*Chairperson*)**
All India Food Processors Association, (India)	MS SHREYA PANDEY SHRI KRISHNA KUMAR JOSHI (*Alternate*)
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Organization	*Representative(s)*
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VR Food Tech Pvt Ltd	DR ASHLESH PARCHURE DR N. RAM (*Alternate*)
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SCIENTIST 'B'/ASSISTANT DIRECTOR
(FOOD AND AGRICULTURE), BIS

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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